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Description

TECHNICAL FIELD

5 The present invention relates to biodegradable fabric softening compositions which exhibit excellent hydrolytic stability upon storage. More particularly, the invention relates to aqueous dispersions of biodegradable quaternary ammonium compounds suitable as rinse-added fabric softener compositions, which are formulated at a very specific pH range in order to ensure maximum hydrolytic stability.

10 BACKGROUND OF THE INVENTION

Rinse-added fabric softener compositions are well-known. Typically, such compositions contain a water-insoluble quaternary-ammonium fabric softening agent. Commercially available fabric softening compositions are basically aqueous dispersions of the water-insoluble quaternary compounds. Quaternary ammonium compounds with long chain alk(en)yl groups interrupted by carboxy groups (i.e., biodegradable quaternary ammonium) are known, from e.g. FR-A-1.593.921. Concentrated softening compositions containing such biodegradable quaternary ammonium are disclosed in EP-A-0 040 562.

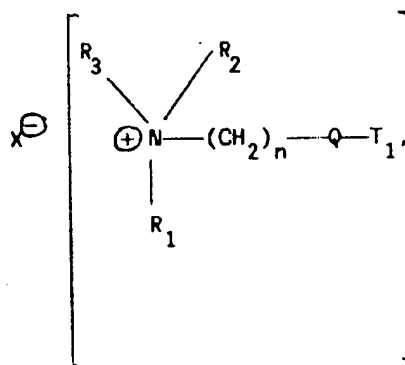
However, since these compounds are intended to be marketed as aqueous dispersions, and since the biodegradable quaternary ammoniums are more subject to hydrolysis than the conventional (DTDMAC-type) cationic softening agents, such biodegradable softening compositions can encounter hydrolytic stability problems upon prolonged shelf storage.

It is therefore an object of the present invention to provide aqueous softening compositions containing biodegradable quaternary ammonium compounds, which are sufficiently shelf stable.

Indeed, it has been found that, by keeping the pH of the compositions herein in a certain range, lower than what is currently used in rinse-added softening compositions, excellent hydrolytic stability is ensured on prolonged shelf storage.

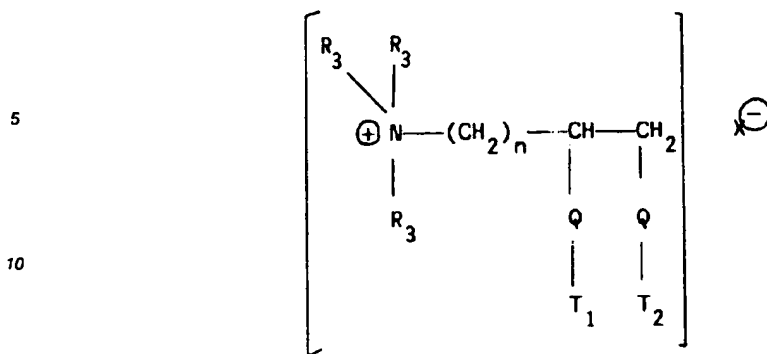
SUMMARY OF THE INVENTION

30 The present invention relates to aqueous fabric softening compositions containing from 1% to 80%, preferably from 2% to 29%, by weight, of a biodegradable quaternary ammonium compound of the formula :



(I)

or

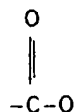


(II)

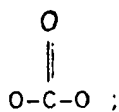
Q is



or



35 or

R₁ is (CH₂)_n-Q-T₂ or T₃ ;R₂ is (CH₂)_n-Q-T₄ or T₅ or R₃ ;45 R₃ is C₁-C₄ alkyl ;T₁, T₂, T₃, T₄, T₅ are (the same or different) C₁₂-C₂₂ alkyl or alkenyl ;

n is an integer from 1 to 4; and

X^e is a softener-compatible anion.

Together with conventional matrix components, and optionally additional softening agents, the pH of the composition being of from 2.5 to 4.2, preferably 3.4 to 4.2, when diluted to a concentration of 0.5% to 1% of the biodegradable quaternary ammonium in water, at 20 °C.

DETAILED DESCRIPTION OF THE INVENTION

55 The biodegradable quaternary ammonium compounds

The biodegradable quaternary ammonium compounds have the formula (I) or (II), above. Preferred compounds are those wherein n = 1 or 2 and R₃ is methyl. Compounds of Formula (I) wherein R₁ is (CH₂)_n

- Q - T₂ and R₂ is (CH₂)_n-Q-T₄ (i.e., quaternary ammonium compounds having three long chains) preferably have at least one unsaturated long chain. Of these, the compounds having all three long chains with one or more double bonds are preferred.

The alkyl, or alkenyl, chain T₁, T₂, T₃, T₄, T₅ must contain at least 12 carbon atoms, preferably at least 16 carbon atoms. The chain may be straight or branched. Unsaturated (alkenyl) chains have been found to impart better rewettability properties to fabrics treated with the softener compositions. Hence, compounds containing such unsaturated chains are preferred in fabric softening compositions intended for use in circumstances where the rewettability properties of the treated fabric is an issue.

Tallow is a convenient and inexpensive source of long chain alkyl and alkenyl material. Compounds wherein T₁, T₂, T₃, T₄, T₅ represents the mixture of long chain materials typical for tallow are particularly preferred.

Specific examples of biodegradable quaternary ammonium compounds suitable for use in the aqueous fabric softening compositions herein include :

- 1) N,N-di(tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride;
- 2) N,N-di(2-tallowoxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride ;
- 3) N,N-di(2-tallowoxyethylcarbonyloxyethyl)-N,N-dimethyl ammonium chloride ;
- 4) N-(2-tallowoxy-2-ethyl)-N-(2-tallowoxy-2-oxo-ethyl) -N,N-dimethyl ammonium chloride;
- 5) N,N,N-tri(tallowyl-oxy-ethyl)-N-methyl ammonium chloride;
- 6) N-(2-tallowoxy-2-oxoethyl)-N-(tallowyl-N,N-dimethyl-ammonium chloride; and
- 7) 1,2-ditallowyl oxy-3-trimethylammoniopropane chloride.

Of these, compounds 1-6 are examples of compounds of Formula (I); compound 7 is a compound of Formula (II).

Particularly preferred is N,N-di(tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride.

Other examples of suitable quaternary ammoniums of formula (I) and (II) are obtained by, e.g.,

- replacing "tallowyl" in the above compounds with, for example, cocoyl, palmoyl, lauryl, oleyl, stearyl, palmityl, or the like;
- replacing "methyl" in the above compounds with ethyl, propyl, isopropyl, butyl, isobutyl or t-butyl;
- replacing "chloride" in the above compounds with bromide, methylsulfate, formate, sulfate, nitrate, and the like.

In fact, the anion is merely present as a counterion of the positively charged quaternary ammonium compound. The nature of the counterion is not critical at all to the practice of the present invention. The scope of this invention is not considered limited to any particular anion.

The compounds herein can be prepared by standard esterification and quaternization reactions, using readily available starting materials.

For example, above compound 1) is prepared by reacting tallow fatty acid with N-methyl-N,N-diethanolamine in xylene at 130°-140° C, whereby water formed in the reaction is removed by azeotropic distillation. The ester thus formed is quaternized with methyl chloride in usual fashion.

Similarly, compound 2) is prepared by reacting imino-diacetic acid with tallow alcohol and subsequent quaternization.

Compound 3) is synthesized by reacting tallow alcohol chloro formate with N-methyldiethanol amine and quaternizing with methyl chloride in usual fashion

The biodegradable quaternary ammonium compounds herein are present at levels of from 1% to 80%, preferably from 2% to 25% by weight of the composition. They can be used in aqueous fabric softening compositions to fully or partially replace conventional, less biodegradable fabric softening ingredients; therefore, the compositions of the invention optionally contain additional softening agents as will be seen hereinafter.

The pH

The pH of the compositions herein is an essential parameter of the present invention. Indeed, it influences the hydrolytic stability of the biodegradable quaternary ammonium compounds, especially in prolonged storage conditions.

The pH, as defined in the present context, is measured in compositions which have been diluted with de-ionized water, at 20° C. The dilution of the compositions whose pH is measured must be such that the biodegradable quaternary ammonium compound is present at a concentration of 0.5% to 1%. For optimum hydrolytic stability of the compositions, the pH, measured in the above-mentioned conditions, must be in the range of from 2.5 to 4.2, preferably 3.4 to 4.2.

The pH of the compositions herein is regulated by the addition of a Bronsted acid.

Examples of suitable acids include the inorganic mineral acids, carboxylic acids, in particular the low molecular weight (C_1 - C_5) carboxylic acids, and alkylsulfonic acids. Suitable inorganic acids include HCl, H_2SO_4 , HNO_3 and H_3PO_4 . Suitable organic acids include formic, acetic, methylsulfonic and ethylsulfonic acid. Preferred acids are hydrochloric, phosphoric, formic, methylsulfonic acid, and benzoic acids.

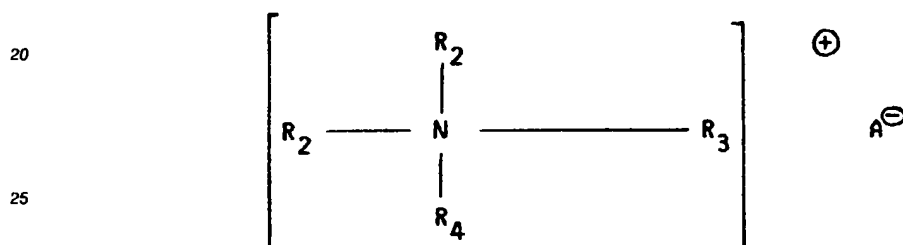
- 5 Fully formulated fabric softening compositions preferably contain, in addition to the biodegradable quaternary ammonium compound of Formula I or II herein, one or more of the following optional ingredients:

Conventional quaternary ammonium salt

- 10 As mentioned before, the biodegradable compounds may be used as a partial replacement of conventional fabric softening active materials, in which case the fabric softening composition further comprises from 2% to 25% of a conventional di(higher alkyl) quaternary ammonium softening agent.

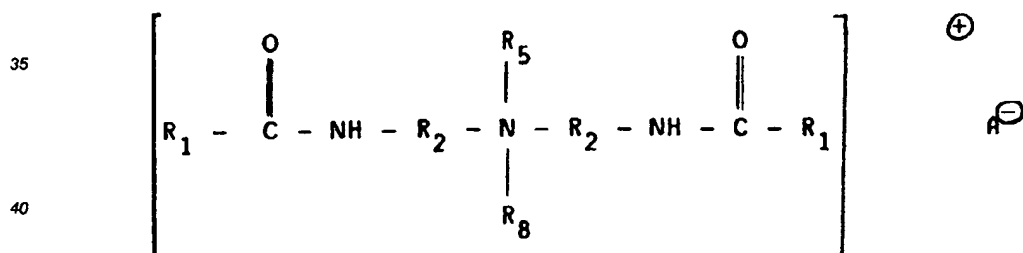
By "higher alkyl" as used in the context of the quaternary ammonium salts herein is meant alkyl groups having from 8 to 30 carbon atoms, preferably from 11 to 22 carbon atoms. Examples of such conventional quaternary ammonium salts include

- (i) acyclic quaternary ammonium salts having the formula:



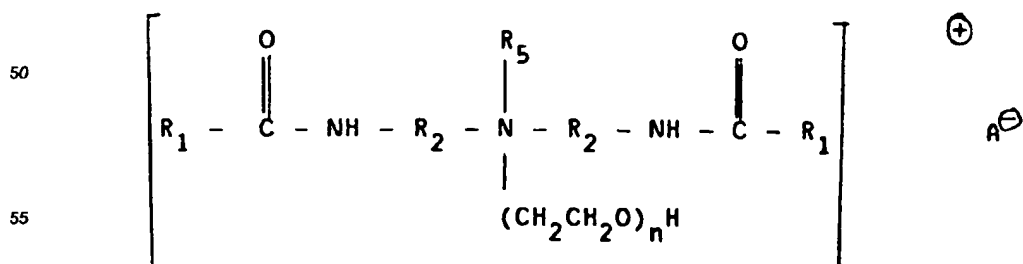
wherein R_2 is an acyclic aliphatic C_{15} - C_{22} hydrocarbon group. R_3 is a C_1 - C_4 saturated alkyl or hydroxyalkyl group, R_4 is selected from R_2 and R_3 , and A is an anion.

- (ii) diamido quaternary ammonium salts having the formula:



wherein R_1 is an acyclic aliphatic C_{15} - C_{21} hydrocarbon group, R_2 is a divalent alkylene group having 1 to 3 carbon atoms, R_5 and R_8 are C_1 - C_4 saturated alkyl or hydroxyalkyl groups, and A^+ is an anion:

- (iii) diamido alkoxyated quaternary ammonium salts having the formula:



wherein n is equal to 1 to about 5, and R₁, R₂, R₅ and A⁶ are as defined above:
(iv) quaternary imidazolinium compounds.

Examples of Component (i) are the well-known dialkyldimethylammoniums salts such as ditallowdimethylammonium chloride, ditallowdimethylammonium methylsulfate, di(hydrogenated tallow) dimethylammonium chloride, dibehenyl dimethylammonium chloride.

Examples of Component (ii) are methylbis(tallowamidoethyl) (2-hydroxyethyl) ammonium methylsulfate and methylbis(hydrogenated tallowamidoethyl) (2-hydroxyethyl) ammonium methylsulfate wherein R₁ is an acyclic aliphatic C₁₅-C₁₇ hydrocarbon group, R₂ is an ethylene group, R₅ is a methyl group, R₈ is a hydroxyalkyl group and A is a methylsulfate anion; these materials are available from Sherex Chemical Company under the trade names Varisoft (R) 222 and Varisoft (R) 110, respectively.

Examples of (iv) are 1-methyl-1-tallowamino-ethyl-2-tallowimidazolinium methylsulfate and 1-methyl-1-(hydrogenated tallowamidoethyl)-methylsulfate.

Typically, the weight ratio rapidly biodegradable: conventional quaternary ammonium compound is in the range from 1:10 to 10:1

Cation-active amines

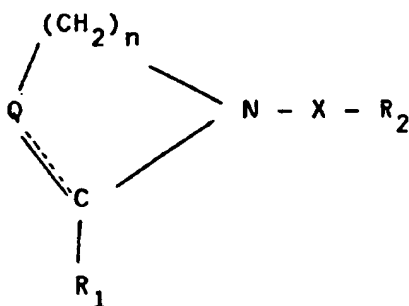
The compositions herein optionally comprise cation-active amines, namely primary, secondary and tertiary amines having, at least, one straight-chain organic group of from 12 to 22 carbon atoms. Preferred amines of this class are ethoxyamines, such as monotallow-dipolyethoxyamine, having a total of 2 to 30 ethoxygroups per molecule. Suitable are also diamines such as tallow-N,N', N'-tris (2-hydroxyethyl)-1,3-propylenediamine, or C₁₆-18-alkyl-N-bis(2-hydroxy-ethyl)amines.

Examples of the above compounds are those sold under the trade name GENAMIN C, S, O and T, by Hoechst.

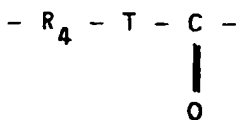
Di-(higher alkyl) cyclic amine

The compositions herein optionally comprise from 1 % to 40 % by weight of the composition of a di-(higher alkyl) cyclic amine of formula IV

Formula (IV)



wherein n is 2 or 3, preferably 2; R₁ and R₂ are, independently, a C₈-C₃₀ alkyl or alkenyl, preferably C₁₁-C₂₂alkyl, more preferably C₁₅-C₁₈ alkyl, or mixtures of such alkyl radicals. Examples of such mixtures are the alkyl radicals obtained from coconut oil, "soft" (non-hardened) tallow, and hardened tallow. Q is CH or N, preferably N. X is



wherein T is O or NR₅, R₅ being H or C₁-C₄ alkyl, preferably H, and R₄ is a divalent C₁-C₃ alkylene group or (C₂H₄O)_m, wherein m is an number of from 1 to 8; or X is R₄.

Optional silicone component

The fabric softening composition optionally contains an aqueous emulsion of a predominantly linear polydialkyl or alkyl, aryl siloxane in which the alkyl groups can have from one to five carbon atoms and may be wholly or partially fluorinated. Suitable silicones are polydimethyl siloxanes having a viscosity at 25 °C in the range from $3,6 \cdot 10^{-1}$ to 360 m²/h (100 to 100,000 centistokes), preferably in the range from 3,6 to 43,2 m²/h (1000 to 12,000 centistokes).

It has been found that the ionic charge characteristics of the silicone as used in the combination are important in determining both the extent of deposition and the evenness of distribution of the silicone and hence the properties of a fabric treated therewith.

Silicones having cationic character show an enhanced tendency to deposit. Silicones found to be of value in providing fabric feel benefits have a predominantly linear character and are preferably polydialkyl siloxanes in which the alkyl group is most commonly methyl. Such silicone polymers are frequently manufactured commercially by emulsion polymerisation using a strong acid or strong alkali catalyst in the presence of a nonionic or mixed nonionic-anionic emulsifier system.

In the present invention, the optional silicone component embraces a silicone of cationic character which is defined as being one of

- (a) a predominantly linear di C₁-C₅ alkyl or C₁-C₅ alkyl, aryl siloxane, prepared by emulsion polymerisation using a cationic surfactant as emulsifier;
- (b) an alpha-omega-di quaternised di C₁-C₅ alkyl or C₁-C₅ alkyl, aryl siloxane polymer or
- (c) an amino-functional di C₁-C₅ alkyl or alkyl aryl siloxane polymer in which the amino group may be substituted and may be quaternised and in which the degree of substitution (d.s.) lies in the range 0.0001 to 0.1, preferably 0.01-0.075.

provided that the viscosity at 25 °C of the silicone is from 100 to 100,000 cs. [$3,6 \cdot 10^{-1}$ to 360 m²/s]

The fabric softening compositions herein may contain up to 10 %, preferably from 0.1 % to 5 %, of the silicone component.

Soil Release Agent

Optionally, the composition herein contain from 0.1 % to 10 %, preferably from 0.2 % to 5 %, of a soil release agent. Preferably, such a soil release agent of the present composition is a polymer. Polymeric soil release agents useful in the present invention include hydroxyether cellulosic polymers, copolymeric blocks of terephthalate and polyethylene oxide or polypropylene oxide, and cationic guar gums, and the like.

The cellulosic derivatives that are functional as soil release agents may be characterized as certain hydroxyethers of cellulose such as Methocel® (Dow); also, certain cationic cellulose ether derivatives such as Polymer JR-125^R, JR-400^R, and JR-30M^R (Union Carbide).

Other effective soil release agents are cationic guar gums such as Jaguar Plus^R (Stein Hall) and Gendrive 458^R (General Mills).

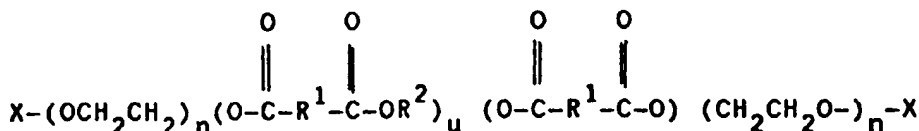
A preferred fabric conditioning composition has a polymeric soil release agent selected from the group consisting of methyl cellulose, hydroxypropyl methylcellulose, or hydroxybutyl methylcellulose, said cellulosic polymer having a viscosity in 2 % aqueous solution at 20 °C of $15 \cdot 10^{-3}$ to 75 Pa.s (15 to 75,000 centipoise).

A more preferred soil release agent is a copolymer having blocks of terephthalate and polyethylene oxide. More specifically, these polymers are comprised of repeating units of ethylene terephthalate and polyethylene oxide terephthalate at a molar ratio of ethylene terephthalate units to polyethylene oxide terephthalate units of from about 25:75 to about 35:65, said polyethylene oxide terephthalate containing polyethylene oxide blocks having molecular weights of from about 300 to about 2000. The molecular weight of this polymeric soil release agent is in the range of from about 5,000 to about 55,000.

Another preferred polymeric soil release agent is a crystallizable polyester with repeat units of ethylene terephthalate units containing 10-15 % by weight of ethylene terephthalate units together with 90-50 % by weight of polyoxyethylene terephthalate units, derived from a polyoxyethylene glycol of average molecular weight 300-6,000, and the molar ratio of ethylene terephthalate units to polyoxyethylene terephthalate units in the crystallizable polymeric compound is between 2:1 and 6:1. Examples of this polymer include the commercially available material Zelcon^R 4780 (from Dupont) and Milease^RT (from ICI).

Highly preferred soil release agents are polymers of the generic formula:

Formula V



in which X can be any suitable capping group, with each X being selected from the group consisting of H, and alkyl or acyl groups containing from 1 to about 4 carbon atoms. n is selected for water solubility and generally is from about 6 to about 113, preferably from about 10 to about 50. u is critical to formulation in a liquid composition having a relatively high ionic strength. There should be very little material in which u is greater than 10. Furthermore there should be at least 20 %, preferably at least 40 % of material in which u ranges from 3 to 5.

The R¹ moieties are essentially 1,4-phenylene moieties. As used herein, the term "the R¹ moieties are essentially 1,4-phenylene moieties" refers to compounds where the R¹ moieties consist entirely of 1,4-phenylene moieties, or are partially substituted with other arylenes or alkarylenes moieties, alkylene moieties, alkenylene moieties, or mixtures thereof. Arylene and alkarylene moieties which can be partially substituted for 1,4-phenylene include 1,3-phenylene, 1,2-phenylene, 1,8-naphthylene, 1,4-naphthylene, 2,2-biphenylene, 4,4-biphenylene and mixtures thereof. Alkylene and alkenylene moieties which can be partially substituted include ethylene, 1,2-propylene, 1,4-butylene, 1,5-pentylene, 1,6-hexamethylene, 1,7-heptamethylene, 1,8-octamethylene, 1,4-cyclohexylene, and mixtures thereof.

For the R¹ moieties, the degree of partial substitution with moieties other than 1,4-phenylene should be such that the soil release properties of the compound are not adversely affected to any great extent. Generally, the degree of partial substitution which can be tolerated will depend upon the backbone length of the compound, i.e., longer backbones can have greater partial substitution for 1,4-phenylene moieties. Usually, compounds where the R¹ comprise from about 50 to 100 % 1,4-phenylene moieties (from 0 to about 50 % moieties other than 1,4-phenylene) have adequate soil release activity. For example, polyesters made according to the present invention with a 40:60 mole ratio of isophthalic (1,3-phenylene) to terephthalic (1,4-phenylene) acid have adequate soil release activity. However, because most polyesters used in fiber making comprise ethylene terephthalate units, it is usually desirable to minimize the degree of partial substitution with moieties other than 1,4-phenylene for best soil release activity. Preferably, the R¹ moieties consist entirely of (i.e., comprise 100%) 1,4-phenylene moieties, i.e. each R¹ moiety is 1,4-phenylene. (Irrespective of the mechanism of action, it is surprising that the soil release polymers do show excellent benefits on fabrics other than polyester fabrics and the compositions herein are designed to clean all manner of fabrics and textiles.)

For the R² moieties, suitable ethylene or substituted ethylene moieties include ethylene, 1,2-propylene, 1,2-butylene, 1,2-hexylene, 3-methoxy-1,2-propylene and mixtures thereof. Preferably, the R² moieties are essentially ethylene moieties, 1,2-propylene moieties or mixture thereof. Inclusion of a greater percentage of ethylene moieties tends to improve the soil release activity of the compounds. Surprisingly, inclusion of a greater percentage of 1,2-propylene moieties tends to improve the water solubility of the compounds.

For this invention, the use of 1,2-propylene moieties or a similar branched equivalent is desirable for incorporation of any substantial part of the soil release component in the liquid fabric softener compositions. Preferably, from about 75 % to about 100 %, more preferably from about 90 % to about 100 % of the R² moieties are 1,2-propylene moieties.

The value for each n is at least about 6, but is preferably at least about 10. The value for each n usually ranges from about 12 to about 113. Typically, the value for each n is in the range of from about 12 to about 43.

A preferred process for making the preferred soil release component comprises the step of extracting a polymer having a normal distribution in which a substantial portion comprises a material in which u is greater than 6 with essentially anhydrous ethanol at low temperatures, e.g. from about 10 °C to about 15 °C. The ethanol soluble fraction is substantially free of the longer polymers.

Organic solvent

The compositions of the present invention can be formulated without the use of any organic solvent. However, the presence of organic solvents (for example, low molecular weight, water miscible aliphatic alcohols,) does not harm the storage stability, the viscosity, or the softening performance of the composi-

tions of this invention.

Typically, quaternary ammonium salts will be obtained from a supplier of bulk chemicals in solid form or as a solution in an organic solvent, e.g., isopropanol. There is no need, whatsoever, to remove such a solvent in making the compositions of this invention. Indeed, additional solvent may be added, if this is deemed desirable.

Optional Nonionics

The compositions optionally contain nonionics as have been disclosed for use in softener compositions. Such nonionics and their usage levels, have been disclosed in U.S. Patent 4.454.049, issued June 12, 1984 to Mac Gilp et al.

Specific examples of nonionics suitable for the compositions herein include glycerol esters (e.g., glycerol monostearate), fatty alcohols (e.g., stearyl alcohol), and alkoxyated fatty alcohols. The nonionic, if used, is typically used at a level in the range of from 0.5 - 10 % by weight of the composition.

Other Optional Ingredients

In order to further improve the stability of the compositions herein, and further adjust their viscosities, these compositions can contain relatively small amounts of electrolyte. A highly preferred electrolyte is CaCl_2 .

The compositions herein can optionally contain other ingredients known to be suitable for use in textile softeners. Such adjuvants include perfumes, preservatives, germicides, colorants, dyes, fungicides, stabilizers, brighteners and opacifiers. These adjuvants, if used, are normally added at their conventional levels. However, in the case of composition ingredients utilized for a fabric treatment effect, e.g., perfumes, these materials can be added at higher than normal levels, corresponding to the degree of concentration of the product.

EXAMPLE 1

The following procedure was used to determine the hydrolytic stability of the compositions.

A melt of N-N-di(tallowoyl-oxy-ethyl)-N-N-dimethyl ammonium chloride (1) (about 65 °C) was injected into a waterseat (de-ionized water) of approximately 60 °C while mixing with a baffled stirrer. The pH of finished products (at 20 °C) was varied by adding Hydrochloric Acid or Sodium Hydroxyde to the waterseat prior to injection.

Hydrolytic stability.

The amount of (1) was determined by an CatSO3 titration (complexation with LAS) immediately after making. This amount was in good agreement with the theoretical amount present. Result from CatSO3 titrations were in good agreement with results from titrations with Potassium Hydroxyde before and after saponification (this is the classical method to determine estervalues). The CatSO3 titration was preferred because it allowed more reliable and precise end-point determination. CatSO3 titrations were used to determine the amount of non-hydrolysed (1) after various intervals in time.

Results were as follows:

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	% (1)	AGE	PH	Cat SO ₃ Acid		% Recovery
				estimated	found	
5	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	2.85 2.81 2.80 2.81	0.68	0.40 0.36 0.33 0.33	59 53 49 49
10	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	3.13 3.16 3.27 3.13	0.68	0.47 0.45 0.45 0.45	69 66 66 66
15	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	3.39 3.47 3.46 3.30	0.68	0.68 0.68 0.64 0.61	100 100 94 90
20	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	3.88 4.21 4.12 3.98	0.68	0.69 0.69 0.69 0.69	101 101 101 101
25	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	4.23 4.21 4.13 3.81	0.68	0.72 0.72 0.72 0.70	106 106 106 103
30	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	4.40 4.50 4.42 3.80	0.68	0.71 0.66 0.64 0.51	104 97 94 75
35	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	5.36 4.70 4.40 3.99	0.68	0.65 0.62 0.56 0.54	96 91 82 79
40	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	5.97 5.31 4.98 4.72	0.68	0.57 0.56 0.53 0.53	84 82 78 78
45	5.8	fresh 13 days at Amb. 24 days at Amb. 6 weeks at Amb.	7.51 6.64 6.19 5.82	0.68	0.54 0.53 0.50 0.48	79 78 74 71

The above results clearly show the criticality of pH for hydrolytic stability, and shows the excellent results obtained in the preferred pH range of 3.4 to 4.2.

The following shelf-stable compositions according to the invention are prepared as described in Example 1.

Examples II - VII

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	Ex II	Ex.III	Ex. IV	Ex. V	Ex. VI	Ex. VII
5 DTOEDMAC 1)	14%	2%	10%	16%	20%	8%
DTDMAC 2)	-	4%	10%	-	-	-
Amine 3)	-	2%	-	6%	-	2%
PDMS 4)	-	1%	-	0.5%	0.5%	-
GMS 5)	-	0.5%	1%	-	0.5%	-
SRP 6)	-	-	0.5%	-	0.5%	-
10 Perfume	0.8%	0.5%	0.8%	0.7%	0.8%	0.3%
HCl to pH	3.8	3.8	3.6	3.8	3.6	3.8
Minors 7) & water	balance					

1) N,N-di(2-tallowyloxy-2-oxo-ethyl)-N,N-dimethylammonium chloride

2) ditallowdimethylammonium chloride

3) 1-tallowamidoethyl-2-tallowimidazoline or monotallowdipolyethoxyamine

4) polydimethylsiloxane, having a viscosity of 2,88 m²/h (800 centistokes)

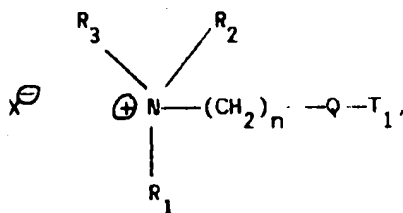
5) glycerylmonostearate

6) soil release polymer of Formula V herein

7) CaCl₂, dye, bactericide.

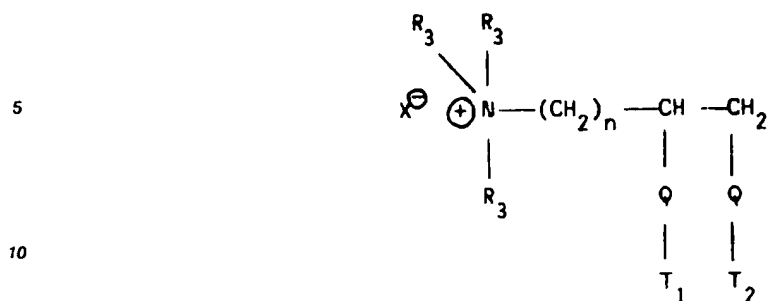
Claims

1. An aqueous fabric softening composition with excellent hydrolytic stability on storage, containing from 1% to 80% of a biodegradable quaternary ammonium softening agent of the formula :



(I)

or



15 (II)

20 Q is



or



35 or



R₁ is (CH₂)_n-Q-T₂ or T₃ ;

45 R₂ is (CH₂)_n-Q-T₄ or T₅ or R₃ ;

R₃ is C₁-C₄ alkyl ;

T₁, T₂, T₃, T₄, T₅ are (the same or different) C₁₂-C₂₂ alkyl or alkenyl ;

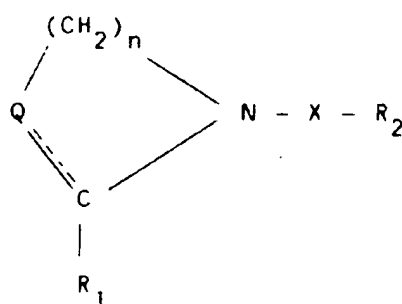
50 n is an integer from 1 to 4; and

X[⊖] is a softener-compatible anion, and, if desired, conventional matrix components and additives, characterized in that the composition has a pH, at 20°C, of from 2.5 to 4.2 upon dilution, in de-ionized water, to a concentration of 0.5% to 1% of said biodegradable quaternary ammonium.

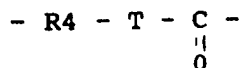
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2. A composition in accordance with Claim 1 wherein said pH is from 3.4 to 4.2.

3. A composition in accordance with claims 1 and 2 wherein the biodegradable quaternary ammonium softening agent is N,N-di(tallowoyl-oxy-ethyl) N,N-dimethyl ammonium chloride.
4. An aqueous fabric softening composition in accordance with claim 1 comprising from 2% to 25% of the biodegradable quaternary ammonium compound.
5. A composition according to claim 1 or 2, further comprising from 2% to 25% of a conventional softening active compound selected from the group of quaternary ammonium salts having at least one acyclic aliphatic C₁₅-C₂₂ hydrocarbon group, and the composition having a weight ratio biodegradable : conventional quaternary ammonium compound in the range from 1:10 to 10:1.
6. A composition according to any one of the preceding claims which further comprises from 1% to 40% of a di-(higher alkyl) cyclic amine of formula:

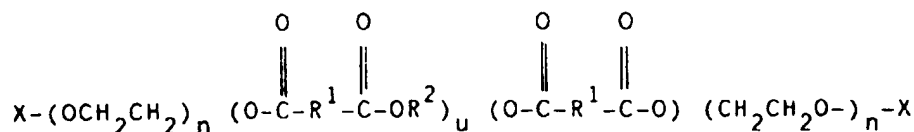


wherein n is 2 or 3, preferably 2; R₁ and R₂ are, independently, a C₈-C₃₀ alkyl or alkenyl, or mixtures of such alkyl radicals. Q is CH or N, X is



wherein T is O or NR₅, R₅ being H or C₁-C₄ alkyl and R₄ is a divalent C₁-C₃ alkylene group or (C₂H₄O)_m, wherein m is a number of from 1 to 8; or X is R₄.

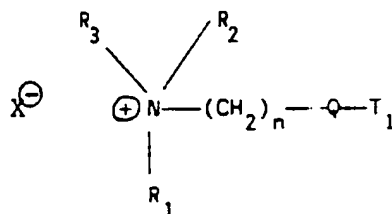
7. A composition according to any one of the preceding claims which further comprises from 0.1% to 10% of a predominantly linear di(C₁-C₅) alkyl or C₁-C₅ alkylaryl siloxane in which the alkyl groups may be partially or wholly fluorinated and which may be substituted with cationic nitrogen groups, the siloxane having a viscosity at 25 °C of at least 100 centistokes and up to 100,000 centistokes. [3.6 10⁻¹ to 360 m²/s]
8. A composition according to claim 7 wherein the siloxane is a polydimethyl siloxane.
9. A composition according to any one of the preceding claims which further comprises from 0.1% to 10% of a soil release agent.
10. A composition according to claim 9 wherein the soil release polymer is of the formula:



wherein R¹ is 1,4-phenylene; R² is 1,2-propylene; X is hydrogen; n is an integer of 6 to 113, u is not greater than 10 with at least 20% of the material having u ranging from 3 to 5.

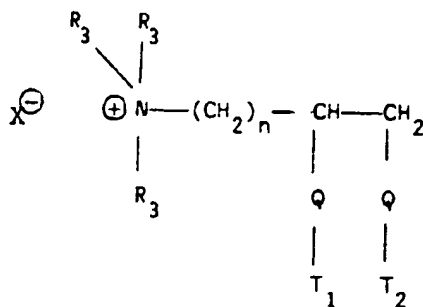
Patentansprüche

1. Wäßrige Gewebeweichmacherzusammensetzung mit hervorragender Hydrolysenbeständigkeit bei der Lagerung, mit einem Gehalt an 1 bis 80 % eines biologisch abbaubaren quaternären Ammoniumweich-
 5 machers der Formel:



(I)

oder



(II)

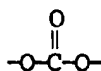
worin
 Q für



oder



oder



steht;
 R₁ für (CH₂)_n-Q-T₂ oder T₃ steht;

R_2 für $(CH_2)_n-Q-T_4$ oder T_5 oder R_3 steht;

R_3 C_1-C_4 -Alkyl bedeutet;

T_1, T_2, T_3, T_4, T_5 (gleiches oder verschiedenes) $C_{12}-C_{22}$ -Alkyl oder -Alkenyl bedeuten;

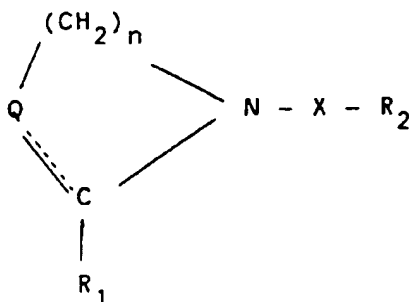
n eine ganze Zahl von 1 bis 4 bedeutet; und

- 5 X° ein Weichmacher-verträgliches Anion darstellt, und gewünschtenfalls an üblichen Matrixkomponenten und an Additiven, dadurch gekennzeichnet, daß die Zusammensetzung bei Verdünnung in entionisiertem Wasser auf eine Konzentration von 0,5 bis 1 % der genannten biologisch abbaubaren quaternären Ammoniumverbindung einen pH-Wert bei 20 °C von 2,5 bis 4,2 aufweist.

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2. Zusammensetzung nach Anspruch 1, worin der pH-Wert 3,4 bis 4,2 beträgt.
3. Zusammensetzung nach den Ansprüchen 1 und 2, worin der biologisch abbaubare quaternäre Ammoniumweichmacher N,N-Di(talgalkyl-oxyethyl)-N,N-dimethylammoniumchlorid ist.
- 15 4. Wäßrige Gewebeweichmacherzusammensetzung nach Anspruch 1, mit einem Gehalt an 2 bis 25 % der biologisch abbaubaren quaternären Ammoniumverbindung.
5. Zusammensetzung nach Anspruch 1 oder 2, mit einem zusätzlichen Gehalt an 2 bis 25 % einer konventionellen weichmachend wirksamen Verbindung, ausgewählt aus der Gruppe der quaternären Ammoniumsalze mit wenigstens einer acyclischen aliphatischen $C_{15}-C_{22}$ -Kohlenwasserstoffgruppe, wobei die Zusammensetzung ein Gewichtsverhältnis von biologisch abbaubarer zu konventioneller quaternärer Ammoniumverbindung im Bereich von 1:10 bis 10:1 aufweist.
- 20 6. Zusammensetzung nach einem der vorstehenden Ansprüche, welche zusätzlich 1 bis 40 % eines Di-(höheralkyl)-cyclischen-Amins der Formel

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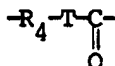


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enthält, worin n für 2 oder 3, vorzugsweise 2 steht; R_1 und R_2 unabhängig voneinander einen C_8-C_{30} -Alkyl- oder -Alkenylrest oder Gemische solcher Alkylreste bedeuten, Q für CH oder N steht, X für

45



r steht,

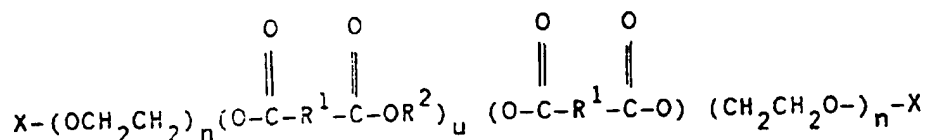
worin T für O oder NR_5 steht, wobei R_5 Wasserstoff oder C_1-C_4 -Alkyl bedeutet, und R_4 eine zweiwertige C_1-C_3 -Alkylengruppe oder $(C_2H_4O)_m$ bedeutet, worin m eine Zahl von 1 bis 8 ist; oder X für R_4 steht.

50

7. Zusammensetzung nach einem der vorstehenden Ansprüche, welche zusätzlich 0,1 bis 10 % eines überwiegend linearen Di(C_1-C_5)alkyl- oder C_1-C_5 -Alkylarylsiloxans enthält, worin die Alkylgruppen teilweise oder vollständig fluoriert sein können und durch kationische Stickstoffgruppen substituiert sein können, wobei das Siloxan eine Viskosität bei 25 °C von wenigstens 100 cSt bis zu 100.000 cSt [$3,6 \times 10^{-1}$ bis 360 m^2/s] aufweist.

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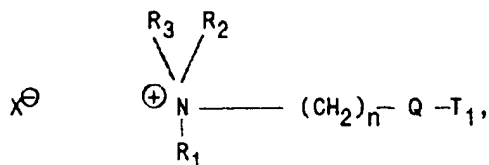
8. Zusammensetzung nach Anspruch 7, worin das Siloxan ein Polydimethylsiloxan ist.
9. Zusammensetzung nach einem der vorstehenden Ansprüche, welche zusätzlich 0,1 bis 10 % eines Schmutzlösemittels enthält.
10. Zusammensetzung nach Anspruch 9, worin das Schmutzlösepolymer die Formel



- aufweist, worin R¹ für 1,4-Phenylen steht; R² 1,2-Propylen darstellt; X Wasserstoff bedeutet; n eine ganze Zahl von 6 bis 113 ist; u nicht größer als 10 ist, wobei wenigstens 20 % des Materials einen Wert u im Bereich von 3 bis 5 aufweisen.

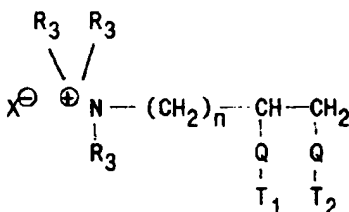
Revendications

1. Composition aqueuse d'assouplissants pour tissus ayant une excellente stabilité hydrolytique au stockage, contenant de 1% à 80% d'un agent assouplissant biodégradable ammonium quaternaire de formules :



(I)

ou



(II)

dans lesquelles :
Q est



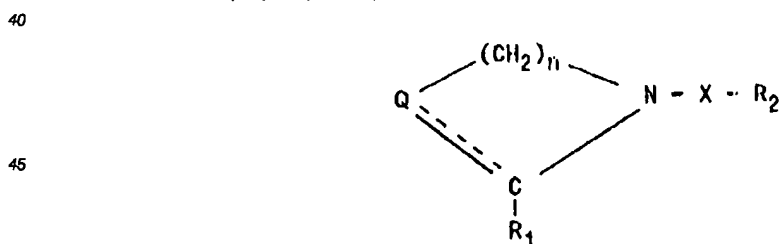
ON



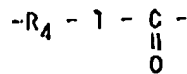
ou



- 15 R₁ est (CH₂)_n-Q-T₂ ou T₃;
R₂ est (CH₂)_n-Q-T₄ ou T₅ ou R₃;
R₃ est un reste alkyle en C₁-C₄;
T₁, T₂, T₃, T₄, T₅ sont (identiques ou différents) un reste alkyle ou alcényle en C₁₂ à C₂₂;
n est un nombre entier de 1 à 4; et
- 20 X^o est un anion compatible avec l'assouplissant, et,
si souhaité, des composants classiques de matrices et des additifs, caractérisée en ce que la composition a un pH, à 20 °C, de 2,5 à 4,2 en dilution, dans l'eau désionisée, à une concentration de 0,5% à 1% dudit ammonium quaternaire biodégradable.
- 25 2. Composition selon la revendication 1, dans laquelle ledit pH est de 3,4 à 4,2.
3. Composition selon les revendications 1 et 2, dans laquelle l'agent assouplissant biodégradable ammonium quaternaire est le chlorure de N,N-di(acyl de sulf-oxy-éthyl) N,N-diméthyl ammonium.
- 30 4. Composition aqueuse d'assouplissants biodégradables pour tissus selon la revendication 1 comprenant de 2% à 25% du composé ammonium quaternaire biodégradable.
5. Composition selon la revendication 1 ou 2, comprenant de plus de 2% à 25% d'un composé actif assouplissant classique choisi dans le groupe des sels d'ammoniums quaternaires ayant au moins un
- 35 groupe hydrocarbure en C₁₅ à C₂₂ aliphatique acyclique, et la composition ayant un rapport pondéral des composés ammonium quaternaire biodégradable/classique dans le plage de 1/10 à 10/1.
6. Composition selon l'une quelconque des revendications précédentes, qui comprend de plus de 1% à 40% d'une di-(alkyl supérieur) amine cyclique de formule



- 50 dans laquelle n est 2 ou 3, de préférence 2;
R₁ et R₂ sont, indépendamment, un reste alkyle ou alcényle en C₈ à C₃₀,
ou des mélanges de tels radicaux alkyles;
Q est un reste CH ou N;
X est un reste



5

dans lequel T est un atome O ou un reste RN₅, R₅ étant un atome H ou un reste alkyle en C₁ à C₄ et R₄ est un groupe alkylène divalent en C₁ à C₃ ou (C₂H₄O)_m, dans lequel m est un nombre de 1 à 8; ou bien X est R₄.

- 10 7. Composition selon l'une quelconque des revendications précédentes, qui comprend de plus de 0,1% à 10% d'un di-alkyl (en C₁ à C₅) principalement linéaire ou alkyl (en C₁ à C₅)-aryl siloxane dans lequel les groupes alkyles peuvent être partiellement ou totalement fluorés et qui peut être substitué avec des groupes azotés cationiques, le siloxane ayant une viscosité, à 25 °C, d'au moins 100 centistokes et allant jusqu'à 100 000 centistokes (100 à 100.000 mm²/s).

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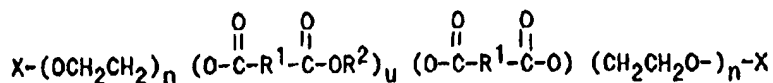
8. Composition selon la revendication 7, dans laquelle le siloxane est un polydiméthyl siloxane.

9. Composition selon l'une quelconque des revendications précédentes, qui comprend de plus de 0,1% à 10% d'un agent de libération de la salissure.

20

10. Composition selon la revendication 9, dans laquelle le polymère de libération de la salissure est de formule:

25



dans laquelle R¹ est le 1,4-phénylène;

30

R² est le 1,2-propylène;

X est un atome d'hydrogène;

n est un nombre entier de 6 à 113;

u n'est pas plus grand que 10 avec au moins 20% du matériau ayant u s'étendant de 3 à 5.

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